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THE PROPERTIES OF PURE NICKEL

RP257

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ABSTRACT

Rather pure electrolytic nickel was further purified by annealing in hydrogen. A study was made of suitable refractory crucibles and of melting conditions to produce extremely pure compact metal. The ingots finally prepared were of a purity of 99.94 per cent. New determinations of many of the physical properties of nickel have been made on the very pure metal thus made available. Data are given on density, crystal-lattice constant, melting point, electrical resistivity, magnetic properties, thermal e. m. f., thermal expansion, hardness, tensile strength, and reflectivity.

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I. INTRODUCTION

In October, 1925, the International Nickel Co., through the courtesy of Dr. Paul D. Merica, assistant to the president, made available to the National Bureau of Standards small amounts of two lots of rather pure electrolytic nickel for use in such determinations of the physical properties of the pure metal as it might prove possible for the Bureau of Standards to undertake.

Preliminary melting experiments were carried out with the less pure of these two lots of materials. Some further purification of the better of the two lots was undertaken, and vacuum fused ingots of this nickel were made. Suitable specimens were prepared for a variety of determinations of physical properties, namely, density, lattice constants, hardness, thermal transformation, melting point, magnetic properties, electrical resistivity, temperature coefficient of electrical resistance, thermal electromotive force, thermal expansion, and reflectivity.

¹ With the collaboration of members of the bureau staff, as indicated in the various sections of the report.

A detailed discussion of the preparation and purity of the materials used in the above determinations is given in the present paper as well as a fairly detailed summary of all of the above work on physical properties. The methods and results involved in certain of the determinations of the properties of nickel are, or will be, described in still greater detail in separate papers.

A great many members of the bureau staff have cooperated in the securing of these data on pure nickel. In addition to those to whom credit is given in the various sections of this report, thanks are especially due to H. C. Vacher, of the division of metallurgy, for the determination of the gases, and to J. A. Scherrer and C. P. Larabee, of the chemistry division, for the determinations of the other impurities of the nickel specimens.

II. MATERIALS

The two lots of cathode nickel, which were in the form of thin flakes of metal, were designated as lot Ni-1-A and lot Ni-2-B. As received they were of the following purity:

	Ni-1-A	Ni-2-B
	<i>Per cent</i>	<i>Per cent</i>
C-----	0.03	0.05
S-----	.005	(¹)
Si-----	.007	.003
Fe-----	.005	.007
Cu-----	.004	.07
Co-----	.013	.01

¹ Uncertain (possibly 0.005 to 0.012).

Some difficulty was experienced in making satisfactory determinations of sulphur in the original cathode nickel, particularly in the metal of lot Ni-2-B. Some analyses indicated no sulphur in Ni-2-B, others gave about 0.005 per cent, while still others showed as high as 0.012. As is shown later, the sulphur found in fused samples of both Ni-1-A and Ni-2-B melted under conditions which should not introduce sulphur, indicated that the actual sulphur content of both lots of nickel was of the order of 0.010 per cent.

TABLE 1.—*Effect of vacuum fusion in various types of crucibles on the composition of electrolytic nickel of lot Ni-2-B*

Melt No.	Type of crucible	Type of furnace	Purity of resulting ingot					
			C	P	S	Si	Fe	Cu
N-1 ¹ -----	Zirconium silicate-----	Arsem-----	<i>Per cent</i> <0.01	<i>Per cent</i> 0.59	<i>Per cent</i> 0.015	<i>Per cent</i> 0.003	<i>Per cent</i>	<i>Per cent</i>
N-2 ² -----	do-----	do-----	<.01	.59	.015	.008	-----	-----
N-3-----	RR alundum and No. 518 alundum cement-----	do-----	-----	.009	.016	.31	-----	-----
N-4-----	Zirconium silicate ³ -----	do-----	-----	.24	.011	.079	-----	-----
N-5-----	Sillimanite porcelain (aluminum silicate)-----	do-----	.015	.005	.008	.01	0.017	-----
N-6-----	C. P. MgO+shellac-----	do-----	.04	<.003	.014	.03	.027	0.028
N-7-----	Same as N-6; second melt-----	do-----	-----	-----	.009	.02	-----	-----
N-8-----	Same as N-6; third melt-----	do-----	.015	-----	.009	.01	.004	-----
N-9-----	New C. P. MgO+shellac-----	High-frequency-----	.03	<.001	.010	.009	-----	-----
N-10-----	Reground C. P. MgO+Shellac-----	Arsem-----	-----	-----	.008	.006	-----	-----
N-11-----	Commercial sintered MgO; inside of crucible fused and glazed under electric arc-----	do-----	-----	-----	.031	-----	-----	-----

¹ The cathode nickel of the crucible charge was a mixture of coarse flakes and the finer as received.

² The nickel of the charge was sifted free of the finer flakes.

³ A different lot of ZrSiO₄ from that used in N-1 and N-2 melts.

III. PRELIMINARY MELTING EXPERIMENTS

The details of the preliminary melting experiments, carried out in order to select the most satisfactory type of crucible and furnace for preparing the final specimens, are given in Table 1. The material used for the crucible charge in all melts from N-1 to N-11, inclusive (Table 1), was the less pure nickel, Ni-2-B. The first eight melts were made in an Arsem vacuum furnace (pressures of 5 to 16 mm of mercury) as were also melts N-10 and N-11. Ingot N-9 was fused in vacuum (pressure of less than 1 mm of mercury) in the high-furnace induction furnace.

The three melts made in zirconium silicate crucibles were all contaminated by phosphorus (N-1, N-2, and N-4), although the zirconium silicate used was the commercial refined grade. The carbon content of all melts was slightly lower than that of the original cathode nickel. This decarburization was noticeable with all types of crucibles, although it was very slight in the first melt in a magnesia-shellac crucible.²

The silicon in the nickel increased very noticeably when the metal was melted in aluminum oxide (N-3) or in zirconium silicate (N-4). The alumina crucible, however, contained some commercial alundum cement (No. 518), which was probably considerably higher in silica than the RR alundum. Increase of silicon in the melt took place after some melts in C. P. MgO crucibles (N-6 and N-7) but appeared to be not necessarily characteristic of all melts in this refractory (N-8, N-9, and N-10). Melts N-7 and N-8 were, respectively, the second and third melts of fresh charges of cathode nickel in the same magnesia crucible. In these cases the solidified ingot did not wet or stick to the magnesia crucible and could be removed after cooling to room temperature by gentle tapping and without injury to the crucible. The remelts were made to determine if appreciable purification of the refractory occurred on continued use, particularly as indicated by an increase in the sulphur content of the nickel ingot.

The most serious contamination in any of these trial melts was that due to sulphur. It has been shown by Merica and Waltenberg³ that remelted electrolytic nickel is rendered almost completely non-malleable by as little as 0.01 per cent sulphur and malleability was, of course, essential in the preparation of the specimens necessary for the present work.

In all of the preliminary melts, N-1 to N-10, regardless of the nature of the crucible, the sulphur content of the resulting ingot was practically 0.01 to 0.02 per cent, whereas the analysis of the original cathode nickel of lot Ni-2-B had shown generally not over 0.005 per cent sulphur. As has been noted before there may be reason to doubt the accuracy of the analytical values for sulphur obtained, even with great pains in the analytical procedure, in the case of this cathode nickel.

A final melt of this lot of nickel (N-11) was made in a crucible of commercial electrically sintered magnesia. The inside surface of this crucible was fused and glazed by spinning the crucible while an arc

² The magnesia-shellac crucibles were made as described by R. F. Mehl, J. L. Whitten, and D. P. Smith, Laboratory Production of Pure Magnesia Ware. *Ind. Eng. Chem.*, **17**, p. 1171, 1925.

³ P. D. Merica and R. G. Waltenberg, Malleability and Metallography of Nickel, B. S. Tech. Papers No. 281; 1925.

was burning between two graphite electrodes inside the crucible. It was known that the commercial sintered magnesia crucibles which had not been glazed inside were prone to contaminate melts with sulphur, but it was thought that possibly a dense glaze on the surface might reduce this contamination and that the glazed surface might be purified in the course of the fusion process. However, the resulting ingot (N-11) contained the most sulphur of any of the ingots of this first series.

TABLE 2.—Effect of hydrogen treatment and of melting in various types of crucibles on the composition of electrolytic nickel of lot Ni-1-A

Melt No.	Type of crucible	Type of furnace	C	Mn	P	Purity of resulting ingot						Ni direct determination
						S	Si	Fe	Cu	Co	Mg	
N-12	Mullite (aluminum silicate)	Arsem.	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent
N-13	Fused Al_2O_3	do	---	---	---	0.01	0.07	---	---	---	---	---
N-14	C. P. MgO—shellac	do	---	---	---	.010	.12	---	---	---	---	---
NH-1	Alundum	Pt.	---	---	---	.011	.03	---	---	---	---	---
NH-2	Fused silica	Nichrome resistance furnace at 1,450° C. in H_2	<0.005	---	---	<.002	.14	---	---	---	---	---
N-15	C. P. MgO — shellac	do	---	---	---	.003	.006	---	---	---	---	---
N-16	C. P. MgO + MgCl_2	Arsem.	.05	---	---	<.002	.02	---	---	---	---	---
N-17	do	do	---	---	---	---	---	---	---	---	---	---
N-18	do	do	---	---	---	---	---	---	---	---	---	---
N-19	do	High-frequency	(¹)	(¹)	(¹)	.004	(¹)	0.024	0.016	0.016	0.003	---
N-20	do	Arsem.	---	---	---	---	---	---	---	---	---	---
N-21	do	do	---	---	---	---	---	---	---	---	---	---
N-22	do	do	---	---	---	---	---	---	---	---	---	---
N-23	do	High-frequency	.005	(¹)	(¹)	.004	.006	.03	.006	.016	(¹)	99.94

¹ Not detected.

IV. MELTS OF THE PURE ELECTROLYTIC NICKEL

Attention was next turned to the purer electrolytic nickel of lot Ni-1-A in the hope that this material would prove lower in sulphur after fusion than had the Ni-2-B nickel. The first three ingots prepared from this material, however, all showed 0.01 per cent sulphur (N-12, N-13, and N-14, Table 2). They were melted, respectively, in crucibles of mullite (aluminum silicate), in fused alumina (RR alundum), and in C. P. magnesia bonded with shellac. The contamination of the melt with silicon from both the aluminum silicate and the fused alumina was excessive and was very considerable even from the C. P. magnesia.

It appeared from the foregoing melts (N-1 to N-14, inclusive), that the electrolytic nickel contained at least 0.01 per cent sulphur in some form in spite of the lower values indicated by analyses of the original cathode metal, for all melts of both lots of nickel contained at least that proportion of sulphur, regardless of the refractory crucible material used. There were also indications that new crucibles of magnesia bonded with shellac tended either to introduce carbon into the melt (N-6 and N-9) or at least to prevent the small, but appreciable, decarburization caused by melting in other oxide crucibles (N-1, N-2, N-5, and N-8). Furthermore, when the carbon in the resulting ingot was of the order of 0.04 per cent (N-6), or from the type of crucible used might be expected to contain carbon to about that amount (N-14), the increase of silicon in the melt even from the C. P. magnesia crucible was very considerable.

It was, therefore, decided to treat the cathode nickel in hydrogen at an elevated temperature (for the removal of both carbon and sulphur from the metal) and to use C. P. magnesia crucibles bonded with a solution containing 2 g of magnesium chloride (MgCl_2 , $6\text{H}_2\text{O}$) in 100 ml of water.⁴

The first hydrogen treatment of nickel was carried out by placing a small charge of metal from lot Ni-1-A in an alundum boat within a porcelain tube and heating to approximately $1,450^\circ\text{C}$. in a platinum resistance furnace. A stream of dry purified hydrogen was passed through the tube during the heating. The temperature actually reached was somewhat higher than had been intended and the nickel sample fused. As might be expected, the silicon content of this fused nickel button was high, namely, 0.14 per cent (NH-1, Table 2), but the sulphur was gratifyingly low. It was less than 0.002 per cent.

The next hydrogen treatment of nickel was carried out by packing rather tightly a 3 or 4 inch length of the middle section of a 1-inch diameter fused silica combustion tube with the cathode nickel and heating this in a stream of hydrogen to $1,050^\circ\text{C}$. in a nichrome resistance furnace. The heating was continued for about one and one-half hours. The metal resulting from this treatment (NH-2) was of quite satisfactory purity; it contained less than 0.005 per cent of carbon, together with 0.003 sulphur and 0.006 silicon. There had thus been a very appreciable improvement as regards carbon and sulphur. A small trial melt of this hydrogen-treated nickel was made in a magnesia-shellac crucible (N-15) in the Arsem furnace

⁴ L. Jordan, A. A. Peterson, and L. H. Phelps, *Refractories for Melting Pure Metals; Iron, Nickel, Platinum*, Trans. Am. Electrochem. Soc., 50, p. 155; 1926.

and produced an ingot entirely satisfactory as regards its sulphur content (less than 0.002 per cent sulphur), but had very clearly been carburized (to 0.05 per cent carbon) by the crucible and had consequently picked up silicon to the extent of 0.02 per cent.

Two small ingots of about 90 g each were next made by melting the hydrogen-treated nickel in the Arsem furnace in magnesia crucibles bonded with magnesium chloride (N-16 and N-17). One of the troubles encountered in melting these crucible charges of rather thin flakes of nickel, which could not be compressed to a very compact charge, was that the nickel charge in the crucible appeared to lag rather far behind the magnesia crucible in attaining the melting temperature. As a consequence, the reaction quite frequently encountered in Arsem furnace work in which the graphite of the supporting shell surrounding the magnesia crucible reacts with the magnesia at elevated temperatures to form magnesium vapors and carbon monoxide gas, got well under way while the nickel in the crucible was still considerably below its melting point. On all colder surfaces close to the heated zones in the Arsem furnace this reaction reverses and a deposit of magnesium oxide and carbon is produced.

There were thus formed thin films of magnesia on the surfaces of many of the nickel flakes and when the nickel melted complete coalescence was prevented by the thin films of magnesia. When the charge was held molten for some time these magnesia films tended to be rejected to the metal-crucible wall interface and to the top surface of the ingot.

In order to eliminate these magnesia films, the two small ingots N-16 and N-17 were etched in HCl, washed, and then melted together in the same type of crucible in the Arsem furnace so as to give a single 180 g ingot, N-18. As a final step, ingot N-18 was remelted in a vacuum, high-frequency induction furnace under a pressure of less than 1 mm of mercury. The magnetic stirring of the liquid metal in this furnace very effectively cleaned out all remaining visible traces of magnesia films.

The complete analysis of the resulting ingot (N-19) showed that carbon, manganese, phosphorus, and silicon were not detectable. The sulphur content was 0.004 per cent, which was believed to be sufficiently low to give a malleable nickel. Copper appeared to have increased over that present in the original cathode nickel, namely, from 0.004 to 0.016 per cent. Cobalt was also 0.016 per cent, about the same as in the original material. Iron had increased noticeably, to 0.024 per cent, and a small amount of magnesium, 0.003 per cent, was present.

A larger ingot of nickel was prepared by this same procedure. Ingots N-20 and N-21, each weighing about 150 g, were cleaned, remelted in the Arsem to give ingot N-22, and a final remelt in the high-frequency furnace was designated as N-23. This ingot was of approximately the same purity as N-19, containing 0.005 per cent carbon, 0.004 sulphur, 0.006 silicon, 0.03 iron, 0.006 copper, and 0.016 cobalt. Manganese, phosphorus, and magnesium were not detected and a direct determination of nickel gave 99.94 per cent. Later analyses for oxygen, hydrogen, and nitrogen in this ingot showed the presence of only 0.001 per cent oxygen and hydrogen, and nitrogen not detected. The complete analysis thus totaled 100.008 per cent.

V. MECHANICAL WORKING AND PREPARATION OF TEST SPECIMENS

Ingot N-23 had an over-all length of 9.5 cm and a central primary pipe opening at the top surface and extending for a little less than 5.5 cm down into the ingot. (Fig. 1.) The upper portion (1.5 to 2.0 cm) of the ingot was removed by a milling machine for samples for chemical analysis. The remainder of the ingot was then cut in two portions such that all the pipe came in the top portion and the bottom (3.5 cm in length) was entirely sound. The portion containing the pipe was then divided longitudinally in two approximately equal parts, designated as B and C. The sound portion at the bottom was designated as D.

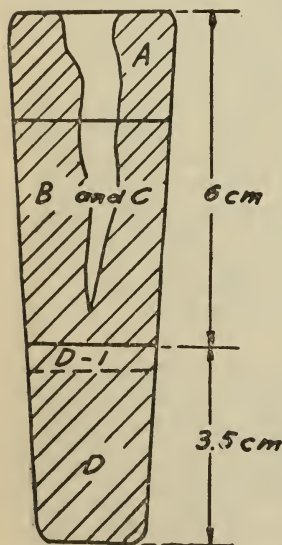


FIGURE 1.—Sections of the split ingot No. 23

The longitudinal sections B and C were mechanically worked to specimens for physical tests. Preliminary to working, the sharp projecting edges in the portion of the pipe in each piece were carefully cut off with a small cold chisel. Section B (or C) was then carefully hammered cold, with frequent annealing, the specimen being manipulated in a grooved anvil block so that the pipe was worked completely to the outer surface of the hammered bar and no seams or laps were formed at the edges of the pipe.

In the early stages of the cold hammering of specimen B sufficiently high annealing temperatures were not employed and a few minute cracks appeared in the metal surface in the pipe cavity. The sections of metal about these cracks were very carefully fused with a small, sharp oxy-hydrogen flame. This specimen was then heated for one-half hour at each of the following temperatures, and scleroscope and Rockwell B hardness numbers were determined. The results indicated that the pure nickel must be annealed at a temperature certainly above 850° C., probably at 950° C., in order to eliminate work hardening.

TABLE 3.—Hardness of nickel

Temperature of heating for one-half hour after cold working (° C.)	Hardness	
	Rockwell B	Scleroscope
As cold worked.....	63-68	12-19
650.....	52-59	8-13
750.....	51-59	8-13
850.....	35-52	7-11
950.....	¹ (15-18)	4-6

¹ Too soft for satisfactory Rockwell B measurements.

Subsequent annealings of specimens B and C were carried out by heating the bar in an oxy-hydrogen flame to about 950° to 1,000° C.

(as judged by eye) for two or three minutes. The nickel was supported on fragments of C. P. magnesia crucibles during such heatings.

The specimen was hand forged to a rod which would enter the 0.330-inch swaging dies. It was then cold swaged, with annealing after each pass, through the 0.330, 0.300, 0.275, and 0.250 inch swaging dies. The bar was not annealed after the 0.250-inch pass. The finished bar weighed 59 g and was 19.5 cm (7.7 inches) long, and 0.65 cm (0.257 inch) in diameter.

The other portion of ingot N-23 (C, fig. 1) was worked in the same manner as B to a bar of very nearly the same size. It differed only in being 0.5 cm (0.2 inch) longer.

Each of the swaged bars (B and C) was then cut into five specimens as shown in Figure 2.

Section D of ingot N-23 was at first held in reserve in case any specimens from the B or C sections proved unsatisfactory, but finally was used as follows: A small disk (marked D-1 in fig. 1) was cut from the top of the original D piece for reflectivity measurements; the remainder was cut in halves longitudinally; one-half was used for melting-point determinations and the other for analyses for oxygen, hydrogen, and nitrogen.

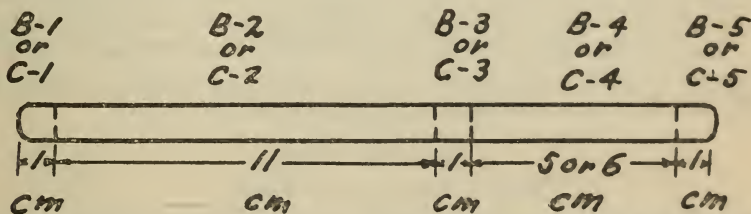


FIGURE 2.—Designation, position, and length of specimens cut from swaged nickel rods

VI. PHYSICAL PROPERTIES

1. DENSITY

(By E. L. Peffer ⁵)

The density of the pure nickel of ingot N-23 was determined (a) in the "as-cast" condition, or rather as quite slowly cooled from the liquid and frozen in very large crystals (secs. B or C and D, fig. 1); (b) as cold-worked after cold swaging from 0.257 to 0.225 inch diameter without annealing; and (c) as annealed by heating at 950° C. for one hour in vacuum. The results are given in Table 4.

TABLE 4.—Density of nickel

Specimen No.	Form of specimen	Condition	Density
			<i>g/cm³</i>
B or C	Part of ingot N-23.....	"As cast".....	8.907 (23° C.)
D	do.....	do.....	8.907 (23° C.)
B-4	Rod 0.225 inch diameter.....	Cold swaged.....	8.901 (25° C.)
C-4	do.....	do.....	8.900 (25° C.)
B-4	do.....	Annealed at 950° C.....	8.903 (25° C.)
C-4	do.....	do.....	8.901 (25° C.)

⁵ Chief, section of capacity and density.

The maximum density 8.907 g/cm^3 was obtained in the very coarsely crystalline ingot metal which had received no mechanical working. The density of the worked metal was slightly less, about 8.901 g/cm^3 either as annealed or after a 30 per cent reduction in area by cold working.

The International Critical Tables⁶ give the density of nickel as 8.90 g/cm^3 at 20°C . The average density of commercial nickel, of purity of about 99.2 per cent, has been given as $8.85 \pm 0.03 \text{ g/cm}^3$.⁷

2. CRYSTAL STRUCTURE AND LATTICE DIMENSIONS

(By R. G. Kennedy, jr.⁸)

Preliminary measurements of the lattice constant of the unit cube (face centered) of nickel were made on a portion of specimen B-2 after this bar had been used for thermal expansion measurements. The dimension of the side of the unit cube was found to be 3.525 Ångströms. The calculated density of nickel, based upon this value for the lattice dimension, is 8.917 g/cm^3 as compared with the determined values of 8.907 "as cast" (large crystals) and 8.901 for the worked specimens.

The length of the side of the unit cube of nickel as given by Clark⁹ is 3.499 Ångströms which gives a calculated density for nickel of about 9.1 g/cm^3 , which is very considerably higher than experimental values. The lattice dimension tabulated by Jeffries and Archer¹⁰ is 3.54 Ångströms which corresponds with a calculated density of only 8.8 g/cm^3 which is appreciably lower than determined values.

3. MELTING POINT

(By H. T. Wensel and Wm. F. Roesser¹¹)

For determination of the melting point of nickel, specimens C-3, B-5, C-5, and one-half of D, all of N-23, were combined to give a single sample of sufficient size for determinations by the crucible method. All of ingot N-19 remaining after the chemical analysis was used for a second or check determination.¹² The specimens were alternately melted and allowed to freeze. Heating and cooling curves were obtained with a precision optical pyrometer sighted into a magnesia tube surrounded by the pure nickel held in a pure magnesia crucible. The crucible and metal were heated or cooled very gradually and uniformly, in vacuum, in a platinum resistance furnace. Very excellent agreement was shown by the data taken by three different observers on the two specimens which represented two ingots of pure nickel. The best value rounded off to the nearest degree for the freezing point of nickel, as shown by this work, is $1,455^\circ \text{C}$. This is 3° higher than the generally accepted value of $1,452^\circ \text{C}$. It is assumed that the temperature of the melting point is the same as that of the freezing point thus obtained.

⁶ I. C. T., 1, p. 104.

⁷ P. D. Merica, Physical and Mechanical Properties of Nickel, Trans. Am. Soc. Steel Treating, 15, p. 1054; 1920.

⁸ Assistant metallurgist, section of optical metallurgy.

⁹ Geo. L. Clark, X-Ray Metallography in 1929, Metals and Alloys, 1, p. 98; 1929.

¹⁰ Jeffries and Archer, The Science of Metals, pp. 4 and 5, McGraw-Hill Book Co.; 1924.

¹¹ Chief and associate physicist, respectively, pyrometry section.

¹² H. T. Wensel and Wm. F. Roesser, The Freezing Point of Nickel as a Fixed Point on the International Temperature Scale, B. S. Jour. Research, 5 (RP 258), p. 1309; December, 1930.

TABLE 5.—*Melting or freezing point of nickel*

Specimen No.	Number of determinations	Average temperature
N-19	9	° C.
N-23	11	1,455
		1,455

As evidence of the absence of any significant contamination of these nickel samples during the many remeltings in the melting point work there is offered the fact that the temperature coefficient of resistance of wires prepared from nickel samples N-19 and N-23 after their use for the melting point determinations was not lower than it had been before the many remeltings in vacuum in the course of the melting point determinations. (See Table 7.)

Analyses for the oxygen, hydrogen and nitrogen content of these two lots of nickel were also made by vacuum fusion before and after the melting-point determinations. A slight increase in oxygen during the remelting was indicated (Table 6), but this increase was without any detectable effect on the melting point as evidenced by the fact that no "drift" in the temperature of the melting point was observed between the first and the last determination on either sample.

TABLE 6.—*Gases in samples of nickel before and after repeated fusions in determinations of the melting point*

Sample No.	Condition	Oxygen	Hydrogen	Nitrogen
		<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>
N-19	Before M. P. determinations.....	0.001	0.0002	(¹)
N-19	After M. P. determinations.....	.008	(¹)	(¹)
N-23	Before M. P. determinations.....	.001	(¹)	(¹)
N-23	After M. P. determinations.....	.015	0.0002	0.004

¹ Not detected.

4. ELECTRICAL RESISTIVITY

(By F. Wenner and F. R. Caldwell¹²)

Specimens B-4 and C-4 of N-23, after being used for density determinations and magnetic measurements, were cold-rolled until long enough to handle with ease in the swaging machines and were then cold swaged to wire 0.079 inch in diameter. This wire was cold drawn to 0.0385-inch diameter through a steel draw plate, except for the last three passes which were through sapphire dies. During this working the metal was annealed in an oxy-hydrogen flame between successive passes through the grooved rolls or through the swaging dies and annealed after every two passes through the drawing dies. Lengths of 120 cm of approximately 1 mm (0.0385-inch) diameter wire of specimens B-4 and C-4 were used for the resistivity measurements, first as cold-drawn through the last two sapphire dies from 0.0435 to 0.0385 inch diameter, and then after having been annealed. The single strand wire, suspended in still air, was annealed at about 800° C. by means of an electric current passed through the wire.

¹² Respectively, chief, section of electrical resistance and assistant physicist, pyrometry section.

Following the determinations of the resistivity, sections of the 0.0385-inch wire from both B-4 and C-4 were drawn to 0.0078-inch diameter for the determination of the temperature coefficient of resistance. In addition to these specimens, there had also been prepared wire specimens of N-19 before and after it was used for determination of the melting point and of N-23 after use for the same determination.

The temperature coefficient of resistance was determined on these additional samples. These values, agreeing within experimental error with those found for the other specimens, showed that no significant contamination occurred while the melting point was being determined.

TABLE 7.—*Electrical resistivity of nickel*

Specimen No.	Condition of metal	Resistivity at 20° C.	Temperature coefficient of resistance 0° to 100° C.
		Microhm-cm	Ohms per ° C. per ohm at 0° C.
B-4	Cold-drawn.....	7.289	-----
C-4	do.....	7.292	-----
B-4	Annealed.....	7.235	0.00667
C-4	do.....	7.237	.00667
N-19	Before M. P. determination.....	-----	.00658
N-19	After M. P. determination.....	-----	.00674
N-23	do.....	-----	.00672

The resistivity of nickel has been given as low as 6.5 microhm-cm¹⁴ and 6.9 microhm-cm¹⁵ at 0° C. This latter value is from Fleming's work¹⁶ and was obtained on a metal whose temperature coefficient between 0° and 100° C. was 0.0061. Copaux¹⁷ gave 0.0066 as the temperature coefficient, more nearly the value found in the present work, but gave the resistivity at 0° C. as 6.4 microhm-cm. Giess and v. Liempt¹⁸ found the temperature coefficient of an "annealed" specimen of very pure nickel to be 0.00667. After further heating of the specimen for 30 minutes at 1,000° C. in vacuum, this value rose to 0.00706.

5. MAGNETIC PROPERTIES

(By R. L. Sanford¹⁹)

Specimen C-2 both as cold worked and after annealing was used for determinations of normal induction, residual induction, and coercive force. The effect of annealing on the normal induction is shown in Figure 3.

The maximum permeability of the cold-worked sample was about 70, while in the annealed specimen the value increased to nearly 1,000. Values for maximum permeability as high as 1,200 were obtained in other specimens of nickel from this same ingot N-23.

For a maximum magnetizing force of 1,000 gilberts per centimeter the residual induction in the cold-worked rod C-2 was 3,280 gauss

¹⁴ P. D. Merica, Physical and Mechanical Properties of Nickel, Trans. Am. Soc. Steel Treat., **15**, p. 1054; 1929.

¹⁵ Int. Crit. Tables, **6**, p. 136.

¹⁶ J. A. Fleming, Electrical Resistivity of Electrolytic Nickel, Proc. Roy. Soc. (London), **66**, p. 50; 1900.

¹⁷ M. H. Copaux, Experimental Researches on Cobalt and Nickel, Ann. Chem. Phys., **6** (8), p. 508; 1905.

¹⁸ W. Giess and J. A. M. v. Liempt, Electric Measurements on Metals of High Purity, Zeit. fur Metallkunde, **17**, p. 194; 1925.

¹⁹ Chief of magnetic section.

with a coercive force of 26.0 gilberts per centimeter. After annealing these values were 5,040 and 6.0, respectively.

Measurements with magnetizing forces up to 300 gilberts per centimeter were made with the magnetic comparator as described by Fischer.²⁰ For magnetizing forces from 100 to 3,000 gilberts per centimeter the modified isthmus method as described by Cheney²¹ was used. There was good agreement between the two methods in the range 100 to 300 gilberts per centimeter common to both.

The saturation value of intrinsic induction $(B-H)_{\infty}$, of a ferromagnetic material is a function of the constitution and is not otherwise dependent upon its condition. The value found for sample C-2, both in the cold-worked condition and after annealing, was 6,150, which agrees with the value given by Droz²² and Hegg.²³ This is also given as the best value by McKeehan²⁴ in his article on "Ferromagnetism."

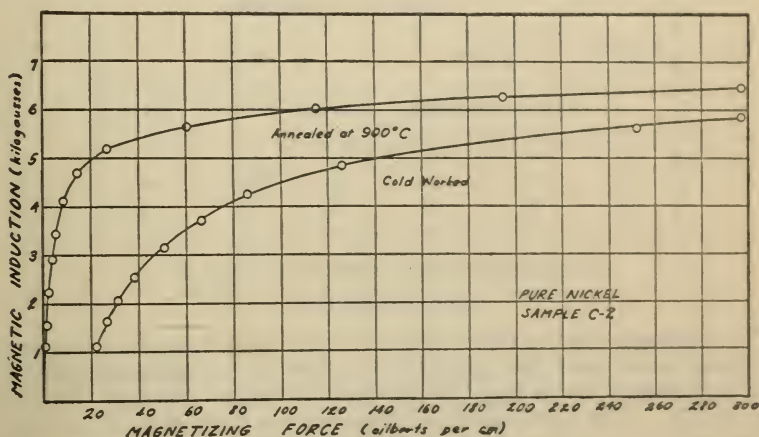


FIGURE 3.—Normal induction of pure nickel

The temperature of the magnetic transformation of nickel from the ferromagnetic to the paramagnetic state is rather indefinite and depends somewhat upon previous history. Various authors give values ranging from 340° to 380° C. The temperature appears to be higher the purer the material.

Several determinations made by the magnetometric method using an astatic magnetometer²⁵ gave on sample C-2 an average value of 380° C. on heating and 371° C. on cooling from approximately 500° C. The individual values ranged from 372° to 387° C. on heating and 367° to 377° C. on cooling. The uncertainty seems to be characteristic of the material and does not depend upon the conditions of test, as no such variation was found for pure iron with the same apparatus under similar conditions.

²⁰ M. F. Fischer, Apparatus for the Determination of the Magnetic Properties of Short Bars, B. S. Sci. Paper No. 458; 1922.

²¹ W. L. Cheney, Magnetic Testing of Straight Rods in Intense Fields, B. S. Sci. Paper No. 361; 1920.

²² Droz, Abram, Determination of Absolute Value of Intensity of Magnetization at Saturation, Arch. des Sci. Phys. et Nat. (4), 29, p. 204; 1910.

²³ Hegg, Felix, A Thermomagnetic Study of Nickel Steel, Arch. des Sci. Phys. et Nat. (4), 29, p. 592; 1910.

²⁴ McKeehan, L. W., Ferromagnetism, J. Frank. Inst., 197, Nos. 5 and 6; 1924.

²⁵ Sanford, R. L., Apparatus for Thermomagnetic Analysis, B. S. Jour. Research, 2 (RP 50); 1929.

6. THERMAL TRANSFORMATION

(By H. C. Cross ²⁶)

The temperature of the thermal transformation of solid nickel was determined by thermal analysis on specimens B-3 and C-3. These specimens, mounted on a platinum/platinum-rhodium thermocouple inclosed in a quartz tube were heated and cooled at a definite rate in a modified Rosenhain furnace.²⁷ A vacuum equivalent to 0.01 to 0.001 mm of mercury was maintained in the tube. Readings every 0.02 mv (approximately 2° C.) were automatically plotted as an inverse-rate curve on a recording chronograph.²⁸ From these the transformation temperatures given in Table 8 were obtained.

TABLE 8.—*Temperature of thermal transformation in nickel. Determined by thermal analysis*

Specimen No.	Temperature of transformation				
	Heating		Cooling		
	Maximum	Ending	Beginning	Maximum	Ending
	° C.	° C.	° C.	° C.	° C.
B-3	353	359	355	{ 351 and 332 }	316
C-3	351	357	357		342

7. THERMOELECTROMOTIVE FORCE

(By F. R. Caldwell ²⁹)

The thermal electromotive force of pure nickel against platinum was determined over the range 0° to 1,100° C. by using nickel wires from specimen C-4 of ingot N-23 and platinum standard Pt 27 of this bureau. The curve shown in Figure 4 has been drawn from data taken at 25° C. intervals over the entire temperature range. The change in the curve in the region of the magnetic transformation of nickel is readily noticeable. The values of the electromotive force at 100° C. intervals with the cold junctions at 0° C. are given in Table 9.

TABLE 9.—*Thermal electromotive force of nickel against platinum*

Temperature of hot junction	E. m. f. ° Pt-Ni	Temperature of hot junction	E. m. f. ° Pt-Ni
° C.	Millivolts	° C.	Millivolts
100	1.485	700	8.105
200	3.105	800	9.350
300	4.590	900	10.695
400	5.450	1,000	12.130
500	6.165	1,100	13.625
600	7.040		-----

* Nickel is thermoelectrically negative to platinum; that is, in a simple thermoelectric circuit formed by these two metals, the current flows from the platinum to the nickel at the cold junction.

²⁶ Formerly assistant metallurgist section of thermal metallurgy.

²⁷ Scott, H., and Freeman, J. R., jr., Use of a Modified Rosenhain Furnace for Thermal Analysis, B. S. Sci. Paper No. 348; 1919.

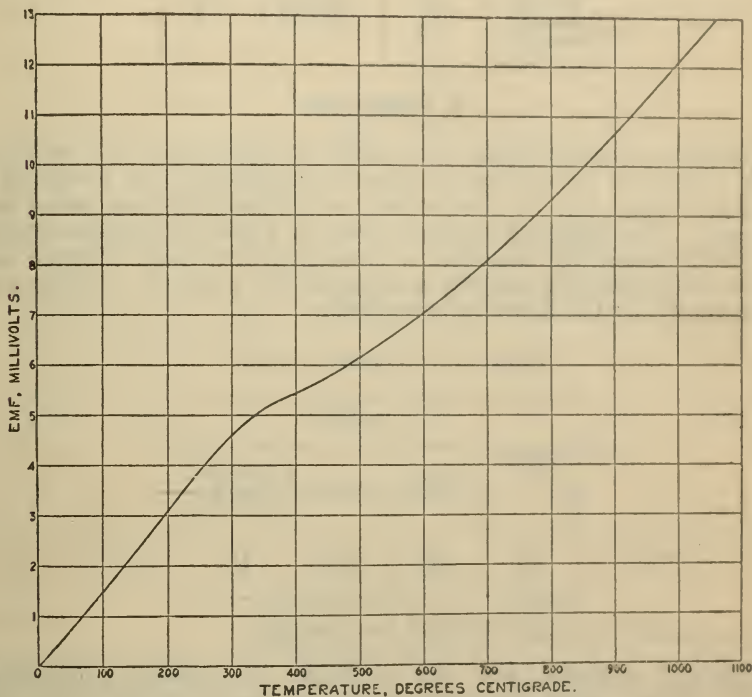
²⁸ French, H. J., A Recording Chronograph for the Inverse Rate Method of Thermal Analyses, B. S. Tech. Paper No. 230; 1923.

²⁹ Assistant physicist, pyrometry section.

8. THERMAL EXPANSION

(By P. Hidnert ³⁰)

The thermal expansion of nickel was determined on specimen B-2 of ingot N-23. This specimen, originally 10 cm (4 inches) long by 0.6 cm (0.24 inch) in diameter as used in the magnetic tests, was annealed and then cold swaged to a diameter of 0.35 cm (0.14 inch) and 31.1 cm (12 inches) long. During the swaging the specimen was annealed after each pass.

FIGURE 4.—*Thermal electromotive force of pure nickel*

Ordinates are the thermal e. m. f. of the couple Pt-Ni; at the cold junction, the current flows from Pt to Ni.

Expansion measurements were taken from 25° to 900° C. A large number of observations were taken between 300° and 400° C. in an attempt to locate a transformation region.

An irregularity in the rate of expansion was shown in the region near 350° C.

³⁰ Associate physicist, section of thermal expansivity.

TABLE 10.—*Thermal expansion of nickel*

Temperature range (in ° C.)	Average coefficient per ° C. ×10 ⁶	Temperature range (in ° C.)	Average coefficient per ° C. ×10 ⁶
25 to 100	13.3	400 to 500	15.9
25 to 300	14.4	500 to 600	16.9
25 to 600	15.5	600 to 700	17.1
25 to 900	16.3	700 to 800	17.7
100 to 200	14.4	800 to 900	18.6
200 to 300	15.4	300 to 600	16.5
300 to 350	17.2	600 to 900	17.8
350 to 400	16.4		

9. HARDNESS

Hardness measurements were made on specimens B-3 and C-3 after they had been used for thermal analysis. The Rockwell B hardness could not be determined satisfactorily as the metal was too soft. The Rockwell numbers given in Table 11 were determined by using a 100 kg load and a one-eighth inch ball. Brinell and Scleroscope numbers are the average of the number of determinations shown by the figure in parentheses.

TABLE 11.—*Hardness of nickel*

Specimen No.	Hardness No.		
	Baby Brinell	Scleroscope	Rockwell 100 kg 1/8-inch ball
B-3	68 (4)	5.0 (3)	42
C-3	78 (4)	5.0 (3)	44

10. TENSILE PROPERTIES

Sufficient material was not available for making satisfactory tensile tests. However, that portion of the 0.139-inch diameter rod remaining after thermal expansion measurements and after cutting a portion for spectrographic tests was broken in tension. The annealed bar broke under a load equivalent to 46,400 lbs./in.² The surface of the bar became so roughened during the flow resulting from the tension loading that the gage marks were entirely obliterated. The elongation was certainly 25 per cent in 2 inches, probably considerably more.

11. REFLECTIVITY

Measurements of the ultra-violet reflectivity of a specimen of this pure nickel (D-1 fig. 1) have been made and already reported by Coblenz and Stair³¹ and compared with the reflectivity of electroplated nickel and of commercial refined nickel. Their results are fully recorded in Figure 5, the curve designated as "C & S vacuum

³¹ Coblenz, W. W., and Stair, R., Reflecting Power of Beryllium, Chromium, and Several Other Metals, B. S. Jour. Research, 2 (RP39), 1929.

fused" representing the results obtained with the pure nickel described in this paper.

VII. SUMMARY

The physical properties of nickel of a purity of 99.94 per cent are summarized in Table 12. This metal was electrolytic nickel fused in vacuum and contained not over 0.001 per cent oxygen.

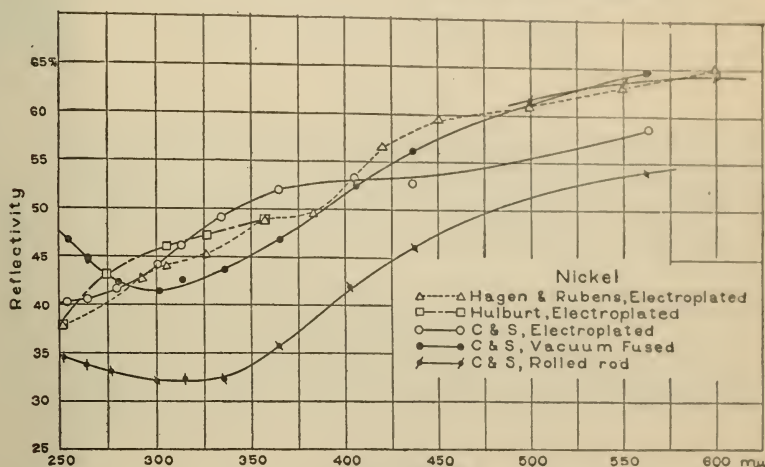


FIGURE 5.—Ultra-violet reflectivity of pure nickel

TABLE 12.—Summary of properties of nickel

Property	Numerical value	Remarks
Density.....	8.907 g/cm ³ at 23° C.....	As cast.
	8.901.....	Worked and annealed.
	8.917.....	Calculated from lattice dimensions.
Length of side of unit cube (face centered).....	3.525 Ångstrom units.....	
Melting or freezing point.....	1,455° C.....	International Temperature Scale.
Electrical resistivity.....	7.236 microhm-cm at 20° C.....	Annealed.
Temperature coefficient of electrical resistance.....	0.0067 ohms per ° C. per ohm at 0° C.....	From 0° to 100° C.
Saturation value of intrinsic induction.....	6,150 gauss.....	
Magnetic transformation.....	370° to 380° C.....	By magnetometer measurements.
Thermal transformation.....	350° C.....	By thermal analysis.
Thermal e. m. f. vs. Pt.....	1.485 mv at 100° C.....	Nickel is thermoelectrically negative to platinum.
	6.165 mv at 500° C.....	
	12.130 mv at 1,000° C.....	
	13.3 × 10 ⁻⁶	
	14.4 × 10 ⁻⁶	
Average coefficient of thermal expansion.....	16.5 × 10 ⁻⁶	25° to 100° C.
	17.8 × 10 ⁻⁶	25° to 300° C.
		300° to 600° C.
		600° to 900° C.
Hardness:		
Baby Brinell.....	6.0 to 78.....	
Scleroscope.....	5.0.....	
Rockwell.....	42 to 44.....	100 kg load; 1/16-inch ball.
Tensile strength.....	46,400 lbs./in. ²	1 specimen only.
Reflectivity.....	See Figure 5.....	

WASHINGTON, October 3, 1930.

